1,126,552

PATENT SPECIFICATION

NO DRAWINGS

1,126,552

Date of Application and filing Complete Specification: 26 May, 1966.
No. 23554/66.
Application made in United States of America (No. 461547) on 4 June, 1965.
Complete Specification Published: 5 Sept., 1968.

Index at acceptance: —B5 B(1R4, 2P2, 12, 17)
Int. Cl.: —D 01 d 5/22

COMPLETE SPECIFICATION

Improvements in the Production of Crimped Staple Fibre

We, FIBER INDUSTRIES, INC., of Box 1414, Charlotte, North Carolina, United States of America, a corporation of Delaware, United States of America, do hereby declare the invention for which we pray that a patent may be granted to us and the method by which it is to be performed to be particularly described in and by the following statement:—

This invention relates to improvements in the production of crimped staple fibre.

In the melt spinning of fibre-forming synthetic polymers especially linear polyesters, asymmetric solidification of the filaments, such as may be achieved, for instance, by directing a stream of cooling gas on one side of the filaments at a point a short distance from the extrusion orifices, yields filaments of unusual properties. The individual filaments as spun exhibit asymmetric birefringence differentials across their diameters, as contrasted with the symmetric differential or absence of differential of a conventional filament, but are straight and indistinguishable visually from conventional filaments. When, however, such asymmetrically solidified filaments are stretched by several times their initial length and then annealed by heating in a relaxed condition, they develop a high degree of three-dimensional crimp such as is not achieved by mechanical crimping, e.g. with a stuffing box or gear-wheel crimper. Staple fibre having this high degree of three-dimensional crimp is of great value for use in making bulky yarns and as filling material for pillows and the like, but the industrial development of the fibre has been hindered by the fact that the annealing operation referred to is expensive because of the time it occupies and has not given results as uniform as could be desired. Thus a period of from three to fifteen minutes or longer is needed to effect annealing in an oven and, in thick layers of fibre on a dryer brattice, movement of fibres within the layers is restricted by surrounding fibres leading to non-uniform crimp development.

According to the invention, crimped staple fibre is produced from filaments made from a themoplastic synthetic polymer and exhibiting asymmetric birefringence differentials across their diameters, by a process which comprises cutting the filaments into staple fibre and feeding ing the staple fibre into a stream of an inert gas heated to a temperature above the second order transition temperature of the polymer and below its softening point, the stream of gas having a sufficient velocity to maintain the fibre in suspension and the fibre being maintained in suspension until it becomes crimped. By the term "inert gas" is meant a gas unreactive with the fibre under the conditions employed.

By means of the invention the time occupied in the annealing operation can be significantly reduced and, at the same time, a product of high uniformity can be obtained. Usually not more than one minute of heating time is needed and in general less than thirty seconds. Preferably the stream of inert gas, e.g. air, nitrogen or carbon dioxide, is caused to lift the staple fibre up a column, the time taken by the fibres in travelling through the column being sufficient to effect the desired annealing. The gas stream may serve the additional function of transporting the staple fibre from its point of production to a packing zone, a screen being used to collect the fibre from the gas stream.

The invention is of particular value in the manufacture of crimped staple fibre from polyethylene terephthalate and will be described more particularly in connection therewith. The invention may, however, be applied to other linear polyesters, e.g. polyhexamethylene terephthalate, including copolymers, e.g. of polyethylene terephthalate with the cor-

[*Price* 4*s.* 6*d.*]

SEE ERRATA SLIP ATTACHED

responding isophtalate or with the 5 (sodium sulpho) isophthalate, and other crystallizable linear polymers of fibre-forming molecular weight, e.g. of intrinsic viscosity between 0.45 and 1.0.

The expression "second order transition temperature", for which the symbol "Tg" is commonly used, is the temperature at which a discontinuity occurs in the curve of a first derivative thermodynamic quantity with temperature. It is correlated with yield temperature and polymer fluidity and can be observed from a plot of density, specific volume, specific heat, sonic modulus or index of refraction against temperature. Tg is sometimes also known as the "glass transition temperature" because it is the temperature below which the polymer exhibits glass-like behaviour and above which the polymer is more rubber-like. Tg for polyethylene terephthalate is approximately 80°C. and the annealing temperature with this polymer is accordingly above this and is preferably between 140 and 180°C.

The filaments initially used in the process may as already indicated, be produced by melt spinning the polymer to form filaments and quenching the filaments asymmetrically by directing a jet of air against one side of them as they emerge from the spinneret orifice. The filaments, generally in the form of tow may be stretched from 2.5 to 8 times their extruded lengths in a liquid, usually water or an aqueous solution or dispersions of such agents as textile finishes and antistatic agents, at a temperature above Tg but below the boiling point of the liquid. If the liquid has a very high boiling point its temperature is usually kept below 140°C.

Immediately after stretching, the wet filaments are cooled to below Tg, preferably by a stream of inert gas, e.g. air, nitrogen or carbon dioxide. The gas, which is preferably air, is aided in its cooling effect by evaporation as the filaments dry in the gas stream. The filaments are then cut into staple fibres of the desired length, e.g. from 1 to 8 inches, and the fibre may then be fed to an enclosed unit, by gravity, mechanical or pnenumatic means, wherein it is swept up into a heated inert gas stream of such velocity that the fibre is kept in suspension in the gas stream. In the hot gas the fibre is annealed to produce the bulky crimped material. The use of staple fibre is essential in this step since each fibre can relax separately with no interference from adjacent fibres as would be the case with a bundle of continuous filaments, or with a thick layer of cut fibre on a dryer brattice.

The heating unit for annealing can be any pipe or column, preferably vertical, of sufficient size and length to permit sufficient residence time of the fibre. The pipe or column can be made out of metal, glass, quartz or any other suitable construction material.

A preferred method of feeding the staple fibre to the annealing unit is by sucking the fibre into a stream of air at temperatures below 60°C. and passing this stream of air carrying the fibre into the heated main stream of air in the annealing unit. The fibre to air ratio in the annealing unit may range, for instance, from 0.001 to 0.50 pound of fibre per cubic foot of air.

The following Examples illustrate the invention:—

EXAMPLE 1

The polymer used is polyethylene terephthalate containing 0.38% TiO_2 and having an intrinsic viscosity of 0.60, Tg for the polymer being 80°C. The polymer is extruded from a conventional melt spinning device maintained at 295°C. through a spinneret plate containing 252 orifices, each 0.009 inch in diameter and 0.012 inch in length, the orifices being arranged on concentric circles and staggered in the successive circles. The extruded filaments are subjected to a high velocity stream of air emerging from a concentric slotted quench cell located 1.5 inches below the spinneret face. The narrow stream of air directed from a 1/16 inch slot at 3.5 cubic feet per minute across the spun threadline causes asymmetrical cooling. The individual filaments produced when examined under a polarising microscope, exhibit an asymmetric birefringence differential across the filament diameter. The filaments are collected on bobbins at a wind-up speed of 3000 feet per minute and at a throughput to give a yarn of 9 denier per filament.

Yarns from 600 bobbins are combined to form a tow comprising 151,200 filaments which is stretched, using conventional equipment comprising a creel guide arrangement for the yarns, a liquid wetting bath maintained at room temperature, 7 feed rolls, hot liquid sprays and 7 draw rolls. The linear speed of the tow at the draw rolls is 200 feet per minute. The draw ratios and temperatures employed for the hot liquid sprays are given in Table 1. After stretching the tow is cooled with a jet of air, at a flow rate of 24 cubic feet per minute. The jet of air is arranged to impinge on the tow immediately past the draw point which is well localized by the hot liquid spray.

The wetting batch and the hot liquid sprays comprise a 1.5% aqueous dispersion of a textile finish containing primarily triethanolamine and butyl stearate. Excess moisture is squeezed out from the tow and the tow is opened with an air jet, then dried in a circulating air dryer at room temperature for 5 minutes.

The oriented, dry filaments are cut to 3 inch length staple fibre which is fed into a column by an air stream at room temperature. At this point the bundles of fibre are separated and the individual fibres relax and de-

velop full crimp as they enter the hot air stream. The fibre when collected on a screen still exhibits differential birefringence when examined under a polarising microscope.

The air velocity of the combined streams is 800 feet per minute, the mix ratio of the hot/cold air is 2:1, the air temperature is 155°C. at the point of entry and 110°C. at the column outlet and the fibre/air ratio is maintained at 0.025 pound of fibre per cubic foot of air. The outlet air is filtered and recycled through a heat exchanger and into the column inlet.

The results obtained are given in Table 1 in which the per cent crimp contraction is calculated in accordance with the following formula:—

$$\text{Per cent crimp contraction} = \frac{(A-B)}{A} \times 100$$

where

A is the taut length of fibre,

B is the reduced length of the crimped fibre.

The "A" length measurements are carried out on fibres using 0.05 gram per denier load and "B" length using 0.005 gram per denier load.

Table 1

Draw Cooling Medium	Draw Temperature °C.	Draw Ratio	% Crimp Contraction
Air	70	3.0	64
„	70	3.2	61
„	70	3.4	60
„	70	3.6	49
„	80	3.0	66
„	80	3.2	63
„	80	3.4	59
„	80	3.6	47
„	90	3.0	61
„	90	3.2	56
„	90	3.4	50
„	90	3.6	34
None	80	3.0	42
„	80	3.2	40
„	80	3.4	21
„	80	3.6	16

The draw ratio in this specification means the ratio of the speed of the final draw roll to that of the feed rolls.

Besides the crimp contraction measurements given bulk measurements were made on the fully processed, relaxed and carded samples using the following procedure:—

A 60 g sample is carded under standard conditions on a sample carding machine to form an 8 inch wide batt which is wound up on a cylindrical core. The core is removed from the rolled batt which is weighed to the nearest 0.1 g. It is then placed on a horizontal plate mounted to an "Instron" compression cell "C" ("Instron" is a Registered Trade Mark). A circular pressure foot is attached to the crosshead and the batt is subjected to 5 compression cycles at the rate of 2 inches per minute. The applied load between 0.475 and 9.500 pounds. At the end of 5 cycles, at low load, the height of the batt is measured. The bulk value is defined as

$$100 \times \frac{\text{Height of batt in inches}}{\text{Weight of batt in grams}}$$

The data are given in Table 2.

Table 2

Draw Cooling Medium	Draw Temperature °C.	Draw Ratio	Bulk Value
None	80	3.2	4.0
„	80	3.4	4.2
Air	80	3.2	4.7
„	80	3.0	5.1
„	90	3.6	3.3

EXAMPLE 2

The filaments are spun as described above in Example 1 and then drawn in tow form at a draw ratio of 3.2 in a finish bath at 80°C. At a short distance past the draw point the tow is passed through a stream of cold water, then wound up, dried and cut in approximately 3 inch staple fibre. After separation these fibres are fed into the heated air column of Example 1. The bulk value of the anneaued material is 4.15.

EXAMPLE 3

The filaments are spun as described in Example 1 and then drawn in tow form at a draw ratio of 3.2 in a finish bath at 82°C. At a short distance past the draw point a high velocity stream of air is directed transverse to the tow by means of a blade shaped jet. After having been cut into 3.75 inch staple fibre, the material is annealed as described above. This material shows a bulk value of 5.0.

The resulting fibre is 5.2 denier per filament and has a tenacity of 3.2 grams per denier, an elongation of 82%, 12.3 crimps per inch and a crimp contraction of 28.7%.

WHAT WE CLAIM IS:—

1. Process for the production of crimped staple fibre from filaments made from a thermoplastic synthetic polymer and exhibiting asymmetric birefringence differentials across their diameters, which comprises cutting the

filaments into staple fibre and feeding the staple fibre into a stream of an inert gas (as hereinbefore defined) heated to a temperature above the second order transition temperature of the polymer and below its softening point, the stream of gas having a sufficient velocity to maintain the fibre in suspension and the fibre being mantained in suspension until it becomes crimped.

2. Process according to Claim 1, wherein the stream of inert gas is caused to lift the staple fibre up a column of sufficient height for the fibre to undergo crimping during its passage through the column.

3. Process according to Claim 1 or 2, wherein the filaments are made from polyethylene terephthalate and the air or other inert gas is heated to between 140 and 180°C.

4. Process for the production of crimped staple fibre, substantially as described in any of the Examples.

5. Crimped staple fibre whenever produced by any of the processes claimed in the preceding claims.

A. J. BUTTERWORTH,
Chartered Patent Agent,
Brettenham House,
Lancaster Place,
Strand, London, W.C.2.

Printed for Her Majesty's Stationery Office by the Courier Press, Leamington Spa, 1968.
Published by the Patent Office, 25, Southampton Buildings, London, W.C.2, from which copies may be obtained.

1,126,552

PATENT SPECIFICATION

NO DRAWINGS

1,126,552

Date of Application and filing Complete Specification: 26 May, 1966.
No. 23554/66.
Application made in United States of America (No. 461547) on 4 June, 19
Complete Specification Published: 5 Sept., 1968.

Index at acceptance: —B5 B(1R4, 2P2, 12, 17)
Int. Cl.: —D 01 d 5/22

COMPLETE SPECIFICATION

Improvements in the Production of Crimped Staple Fibre

ERRATA

SPECIFICATION No. 1,126,552

Page 3, line 74, *for* "3.4" *read* "3.0"
Page 3, line 88, *for* "anneaued" *read* "annealed"

THE PATENT OFFICE
14 October 1968

are straight and indistinguishable visually from conventional filaments. When, however, such asymmetrically solidified filaments are stretched by several times their initial length and then annealed by heating in a relaxed condition, they develop a high degree of three-dimensional crimp such as is not achieved by mechanical crimping, e.g. with a stuffing box or gear-wheel crimper. Staple fibre having this high degree of three-dimensional crimp is of great value for use in making bulky yarns and as filling material for pillows and the like, but the industrial development of the fibre has been hindered by the fact that the annealing operation referred to is expensive because of the time it occupies and has not given results as uniform as could be desired. Thus a period of from three to fifteen minutes or longer is needed to effect annealing in an oven and, in thick layers of fibre on a dryer brattice, movement of fibres within the layers high uniformity can be obtained. Usually n more than one minute of heating time is neede and in general less than thirty seconds. Pr ferably the stream of inert gas, e.g. ai nitrogen or carbon dioxide, is caused to li the staple fibre up a column, the time take by the fibres in travelling through the colum being sufficient to effect the desired annea ing. The gas stream may serve the addition function of transporting the staple fibre fro its point of production to a packing zone, screen being used to collect the fibre from th gas stream.

The invention is of particular value in th manufacture of crimped staple fibre fro polyethylene terephthalate and will be de cribed more particularly in connection then with. The invention may, however, be applie to other linear polyesters, e.g. polyhexamethy lene terephthalate, including copolymers, e. of polyethylene terephthalate with the co

[*Price 4s. 6d.*]

SEE ERRATA SLIP ATTACHED